FINAL REPORT

FOR A

STUDY OF THE USE OF

VARIOUS METALS FOR

OPTICAL REFLECTING ELEMENTS

FR-2259

(S.O. 751)

Contract No. NAS5-9631

Prepared For

Goddard Space Flight Center Greenbelt, Maryland

AMERICAN OPTICAL COMPANY SPACE DEFENSE DIVISION 4709 Baum Boulevard Pittsburgh, Pennsylvania 15213

ABSTRACT

This is the final report on Contract No. NAS5-9631, a study concerning the use of four aluminum alloys, nickel, beryllium, and Haynes 25 as uncoated optical reflecting elements. During this study, beryllium, nickel, and Haynes 25 were successfully optically polished and figured to $\lambda/4$ and their time, temperature, and stress stability were determined. None of the three aluminums could be optically polished and figured. The "follow-on" study of 304L stainless steel will be covered in a second report.

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FINAL REPORT

FOR A

STUDY ON THE USE OF VARIOUS METALS FOR OPTICAL REFLECTING ELEMENTS

1 INTRODUCTION

This report is to summarize the work performed on NAS5-9631. This contract is for a program to:

- 1. Produce high quality optical surfaces on a variety of unplated, metal surfaces. Included are aluminum 2024, aluminum 6061, high purity aluminum (Lurium 5), Haynes 25, beryllium, nickel, and Lockalloy.
- Determine the stability of the resulting pieces as affected by time, temperature and stress.
- 3. Determine the reflectance of the surfaces.

The work concerning 304L stainless steel will be reported on at a later date.

2 DISCUSSION

This study can best be discussed in two parts, the polishing and the testing. The polishing study again was conducted in two parts. First, a series of experiments was conducted with a single sample of each metal to determine the best procedures to produce an optical surface. Secondly, this procedure was then used to produce the samples for testing. When all testing samples were polished, the testing was started.

The polishing will be discussed first, by material.

2.1 Polishing

2.1.1 Nickel

The nickel samples were cut from commercially pure (Ni-200) 4-inch diameter bar stock, rough machined, and heat treated as follows:

- a. Charged into a furnace at $1350 \pm 15^{\circ}$ F
- b. Held 30 minutes
- c. Air-cooled

One surface of each sample was Blanchard ground. The rough grinding and fine grinding of the sample piece was done in the conventional manner using the Microgrit (Micro Abrasive Corporation, Westfield, Massachusetts) abrasive series of WCA #30 (particle size of 30 µ), WCA #12 (particle size 12 µ), and WCA #3 (particle size 3 µ). Although the wide spacing of this abrasive series required prolonged grinding with each grit, the savings in clean-up times more than compensated for this, making it the most efficient series of abrasives. A glass grinding tool was ultimately used in preference to a cast iron tool because it decreased the likelihood of It was also found that the grinding tool should be charged scratches. liberally with grinding compound and water; for if the grinding tool were allowed "to go dry", scratches resulted. The polishing was done on 140 tempered Burgundy pitch (Stevenson Brothers, Philadelphia, Pennsylvania) which had been further tempered by heating, The polishing agent used was Lindy "A" (Union Carbide, East Chicago Illinois) in a water-acetic acid mixture. One part of acetic acid to 50 parts of water were used during the preliminary polishing, and concen tration reduced to one part in 120 for final polishing and figuring. It was found that machine polishing reduced sleeking and that handworkin produced these defects.

The nickel samples for testing were prepared for grinding, as was the sample for the polishing experimentation. Rough grinding, fine grinding and polishing were done with the pieces blocked "five around one" on a "pick-up" block. Such a block is prepared by placing a quarter-inch-thick layer of hard pitch (75175 Pitch, Universal Shellac and Supply Company, Brooklyn, New York) on the back of each piece. The pieces are then placed face down on an optical flat with five pieces symmetrically arranged around a central piece. A heated 12-inch diameter, flat tool is then placed on the pitch and is allowed to melt into the pitch. After cooling, all six pieces are held coplaner and, because of the cold flow tendencies of the pitch, the pieces are held without strain. These blocks were worked as a single piece would be, using the polishing techniques used during the polishing experimentation.

2.1.2 Beryllium

The beryllium samples were of Brush Beryllium's S-200 material. All machining and heat treatment were by Brush. After rough machining, the samples were heat treated at $1600 \pm 10^{\circ}$ F for one hour followed by cooling at a rate no greater than 100° F per hour to 200° F. The pieces were then machined to finished size. All pieces so supplied were uniform in thickness to .002 inches and wedged less than .001 inches. Rough and fine grinding was with the WCA #30, WCA #12, and WCA #3 on a glass grinding tool. Again, the grinding tool was charged liberally and not allowed to go dry. Polishing was done on a pitch lap consisting of 16 parts #140 tempered Burgundy pitch to one part #55 tempered Burgundy pitch (both from Stevens Brothers) with Lindy "A" and water. Again all work was by machine because handwork caused sleeks. The resulting surface contained many small pits which appear to be voids in the material itself.

The beryllium samples for testing were prepared for grinding, as was the sample for the polishing experimentation. These pieces were blocked like the nickel samples and ground and polished, using the techniques perfected during the polishing experimentation. A "clean" surface could not be obtained on the block, and the pieces had to be "de-blocked" and finished individually. This poor surface resulted from the difficulty of getting "good contact" between the block and the polishing lap.

2.1.3 Haynes 25

These samples were sawed from a 4-inch diameter bar, rough machined and heat treated as follows:

- a. Charged into a cold furnace
- b. Heated slowly to 1650 + 25° F and held two (2) hours
- c. Furnace cooled at a rate not exceeding 200° F per hour to 200° F and air cooled.

Rough grinding, fine grinding and polishing of the sample was done in the same manner as the beryllium.

The Haynes 25 samples were fabricated using the techniques used for the beryllium using "pick-up" blocks. No difficulties were encountered.

2.1.4 Aluminum 2024, 6061 and Lurium

The 2024 samples were cut from T-4 bar stock, machined to size, and heat treated 12 hours at 375° F. The 6061 samples were cut from bar stock and finish machined. The Lurium samples were cut from 0.100-inch thick rolled sheet. Approximately 450 hours have

been devoted to experimentation in an attempt to find a method of polishing and figuring these materials. Three of our most highly skilled opticians, including the Optical Shop Foreman, have worked at solving this problem without success. Techniques which produce either a clean surface or a good figure have been found, but no technique which produces both has been found. A summary of the materials and techniques tried in various combinations and permutations include:

a. Grinding Compounds

- 1. Micro Abrasive Corporation WCA Compounds
- 2. Garnet in grease
- 3. Garnet in water

b. Lap Materials

- 1. AO Pellon Pad (American Optical Company, Southbridge, Massachusetts)
- 2. AO Pellon Pad coated with paraffin
- 3. AO Plasti-coated polishing pads
- 4. Experimental AO plastic laps
- 5. Pitch of 3 hardnesses
- 6. Pitch coated with beeswax

c. Polishing Compounds

- 1. Lindy A
- 2. Lindy B
- 3. Lindy C
- 4. Barnesite
- 5. Lustrox 1200 (Tizon Chemical Corporation, Flemington, New Jersey)
- 6. Tizox 100 (Tizon Chemical Corporation)

d. Polishing Vehicles

- 1. Water plus varying concentrations of acetic acid
- 2. Water plus varying concentrations of sodium silicate
- 3. Water plus varying concentrations of acetic acid and sodium silicate
- 4. Ethylene glycol

2.1.4.1 Fine Grinding - The most satisfactory grinding technique was the conventional method using Microgrit WCA powders. This material ground as fast or faster than the carborundums, emerys and garnets. The uniformity in particle size of the WCA's, however, speed the grinding operation by decreasing the chances of scratching. The WCA (t) powders, which are treated so as to remain in suspension in the water vehicle and prevent hard packing of the slurry were also tried. This series seemed to produce less grinding action with no improvement in surface finish. Vehicles other than water such as light machine oil, kerosene, vasoline, etc., drastically reduced cutting action with no improvement in surface finish or "polishability". The series chosen in grinding was WCA #30, WCA #12, and WCA #3. Although the wide spacing of this abrasive series required prolonged grinding with each grit, the savings in clean-up times between grits more than compensated for this. It should be noted that there was no evidence of any of the grits being embedded into the metal samples. Well channelled, glass grinding tools seemd preferable to cast iron or brass because such tools decreased the likelihood of scratches. Also, liberal charging with grinding slurry also decreased the chances of scratching. Machine speed and tooling loading were as extreme as possible until the grinding with WCA #3. At this point, machine speeds and tool loads were reduced. Grinding periods with WCA #3 were twice the time required to remove the pits left by the WCA #12.

2.1.4.2 Polishing - The three (3) most successful polishing approaches were:

a. Pellon on a flat tool - for this approach, an unused AO pellon pad (American Optical, Southbridge, Mass.) was glued to the grinding tool used to fine grind the sample. Polishing was started with Lindy-A and water until the pad was "broken-in" and polishing well. At this point, the polisher was unloaded

and the machine was run moderately fast. The polisher was then wetted with a 50%-50% solution of water and acetic acid but with no additional polishing compound. This technique quickly produced a "good shine" with very slight "orange peel". However, the pellon pad breaks down simultaneously as the shine is produced allowing the optician little or no time to produce a figure. If the figure was not satisfactory, refining and polishing with a new pad was necessary. Satisfactory results, if the slight orange peel could be ignored, depend on the luck of arriving at a satisfactory figure simultaneously with the shine and breaking down of the pad.

- b. Soft Pitch for this approach, a conventional pitch lap consisting of 20 parts of #750 pitch to 1 part of 600 pitch (both from Universal Shellac and Supply Company, Brooklyn, New York) was used. The polishing agent was Lindy-B in water at a pH of 9.0. Extremely slow polishing machine speeds and no other load than the weight of the sample produces a good figure without "orange peel". However, the resulting surface was extremely hazy, scattering approximately 30 percent of the incident light. The logical step of switching to Lindy-A to remove the haze was not successful; the pitch "balled" and adhered to the sample. Similar results were produced using this technique with a pitch lap coated with beeswax.
- c. Polishing Cloth several methods similar to those used by metallurgists to prepare samples for microscopic examination were
 tried. These techniques produced moderate to excellent shines
 with little or no orange peel. Figuring, however, was not controllable.

2.1.5 Lockalloy

The techniques previously described which were successful with pure beryllium were used on the lockalloy. Excellent surfaces were produced with the exception of small small pits running in lines or strings. Prolonged polishing, sufficient to remove several thousandths of material opened as many new pits as were polished out. Specific areas of several samples were monitored microscopically as the polishing progressed, confirming this. The conclusion was reached that the pits resulted from voids in the material and the polishing experimentation stopped.

2.2 Testing

Of the various materials only the nickel, beryllium, and the Haynes were judged suitable for testing. Again, the testing will be describe by material.

2.2.1 Nickel

2.2.1.1 Reflectivity - Reflectivity was measured in several ways. Table I shows specular reflectance at the specified wavelengths as measured with a Carey spectrophotometer with a Strong reflectometer attachment. Figure 1 shows the sum of specular reflectance and scatter as measured with G.E.'s reflectometer and integrating sphere. In this measurement, reflectance was compared with a magnesium car bonate block. Also shown for comparison purposes are the values for freshly evaporated aluminum on optically polished glass. Scattering was approximately 2-1/2 percent.

TABLE I
SPECULAR REFLECTANCE IN PERCENT

Wavelength	Sa	ample Pla	ate	Evaporated	Aluminum
mμ	Ве	Haynes 25	, Ni	Measured	Handbook
250	48.0	37.4	33,2	86.6	92.1
300	43.6	44.7	32.5	87.7	92.3
350	41.5	52.0	36.0	87.7	92.5
400	41.5	55.7	43.0	87.7	92.4
450	41.8	58.6	50.8	87.5	92.2
500	42.4	60.3	53.0	87.3	91.8
550	42.6	61.6	56.6	87,2	91.6
600	43.0	62.4	59.0	86.6	91.1
650	43.2	63.7	61.2	85.8	90.3

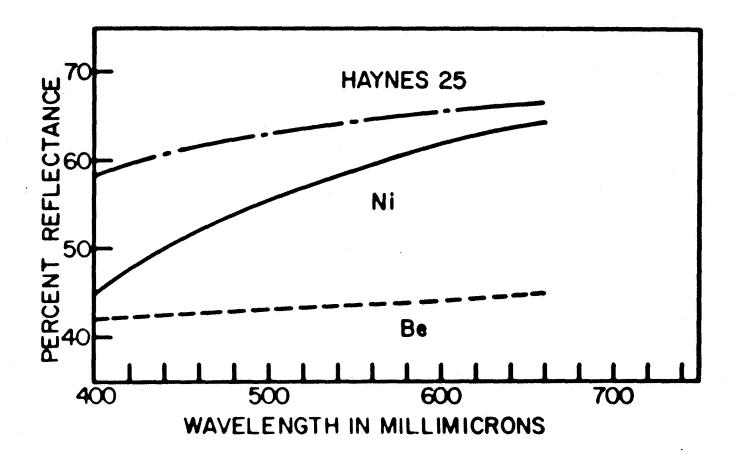


Figure 1. Total Reflectance

2.2.1.2 <u>Time Stability</u> - Time stability testing commenced 9 February 1966 and results through 9 February 1967 are shown in Table II and Figures 2, 3 and 4.

All determinations of flatness were done by photographing Newton's Ring produced between the test piece and a partially aluminized, $\lambda/20$ test flat in 5460 Å green light.

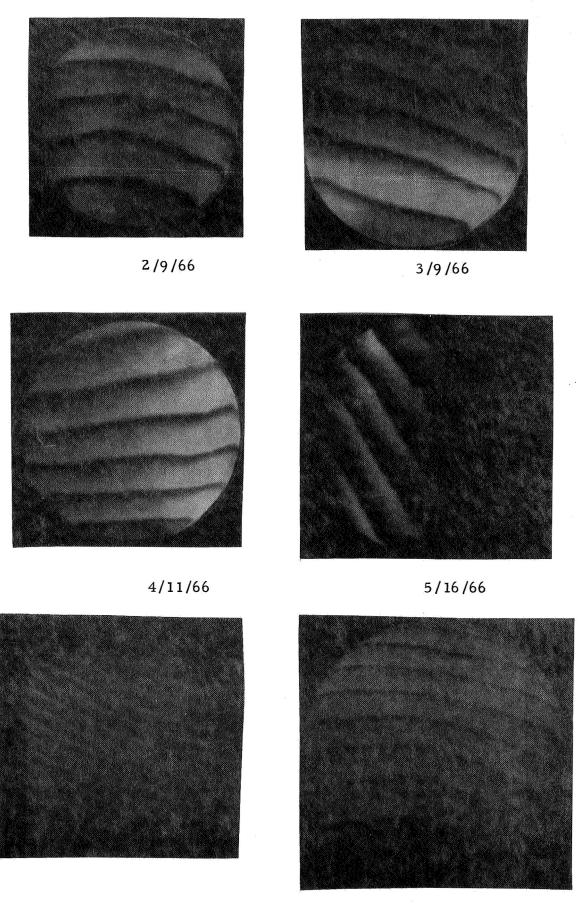
One sample (#4) has remained unchanged (less than $\chi/8$) for a year. One sample (#6) changed during the second month from $\chi/4$ cc flat and $\chi/2$ regular to $\chi/2$ cc flat and $\chi/2$ regular and has remained unchanged since. The third sample (#5) changed from $\chi/2$ cc flat and $\chi/2$ regular to $\chi/2$ cc flat $\chi/2$ regular during the second month; and then changed to $\chi/1$ cc flat and $\chi/2$ regular between third and seventh months; and then changed to $\chi/2$ cc flat and $\chi/2$ regular between the seventh and twelfth months.

2.2.1.3 Cold Cycling Stability - The samples were cold cycled from room temperature in an insulated box using a proprietary solvent and dry ice. All nine samples were cycled simultaneously. Each sample was wrapped in four layers of tissue and then all nine samples were wrapped in four layers of tissue and enclosed in a 4-MIL polyethylene bag. The bag was then immersed in the solvent, and the dry ice added piece by piece so as to lower the temperature slowly. The ultimate temperature reached was -108°F; the entire cycle lasted 20 hours.

Of the three pieces cycled, two were unchanged (less than $\lambda/8$); one (Ni-11) changed from 1/4 λ flat and regular to 1/2 λ flat and regular with 1/4 λ astigmatism. Results are shown in Figure 5.

TABLE II TIME STABILITY TEST RESULTS

				1		2			3			7			12	
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POWER REGI		REGULARITY	POWER	REGULARITY	Po	POWER R	REGULARITY	ā	POWER	REGULARITY	POWER		REGULARITY	POWER		REGULARITY
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λ/2 cc	~	λ/2	λ/2 cc	ς λ/2	λ/2	,2 cc	λ/2		λ/2 cc	λ/2	γ/2	2 CC	λ/2	γ/2	သ	λ/2



9/9/66

5/16/66

Figure 2. Time Stability Testing Of Nickel (Sample No. 4)

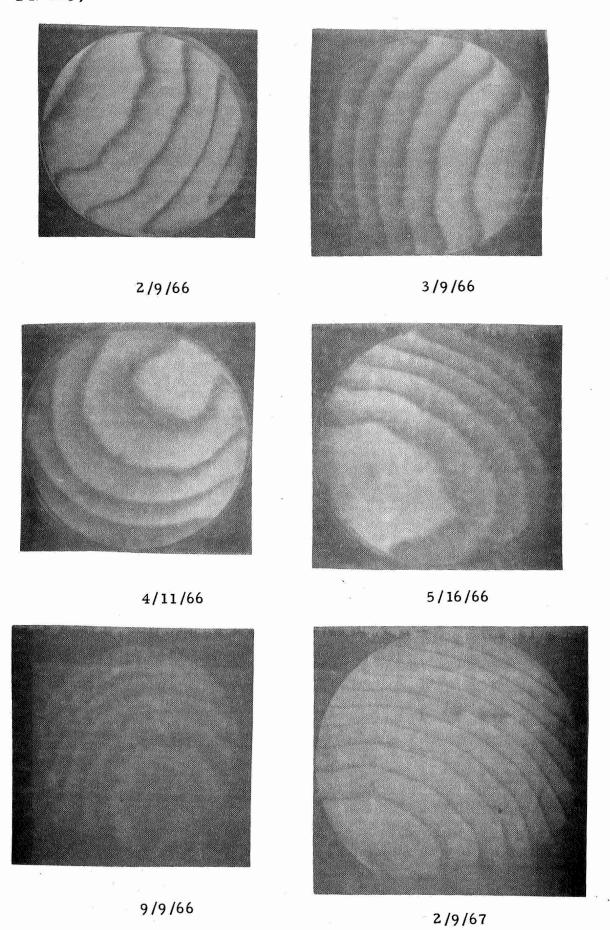


Figure 3. Time Stability Testing of Nickel (Sample No. 5)

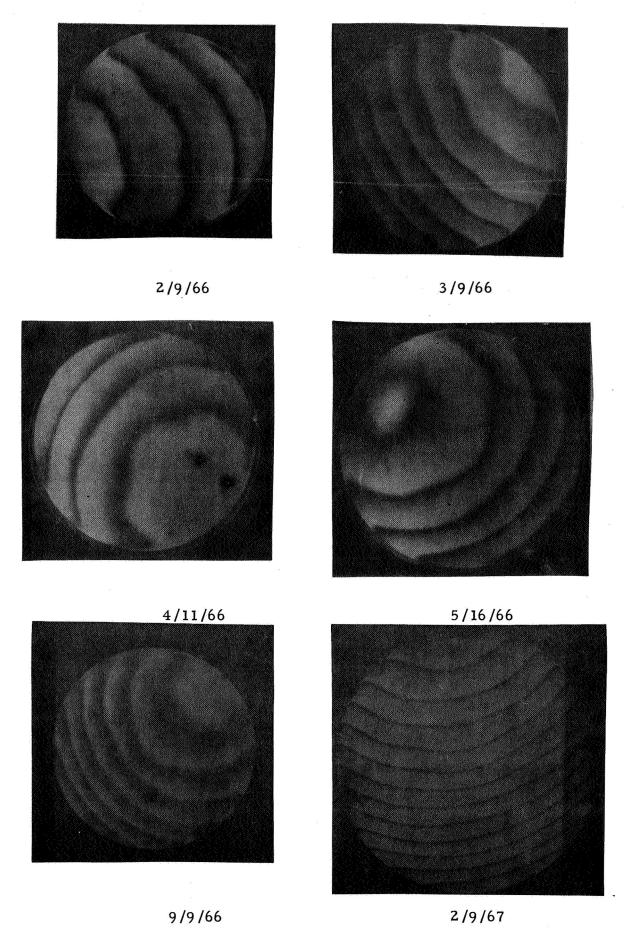


Figure 4. Time Stability Testing of Nickel (Sample No. 6)

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BEFORE FREEZING AFTER FREEZING SAMPLE 10 SAMPLE 11

SAMPLE 12

Figure 5. Cold Cycling Stability Testing Of Nickel Samples

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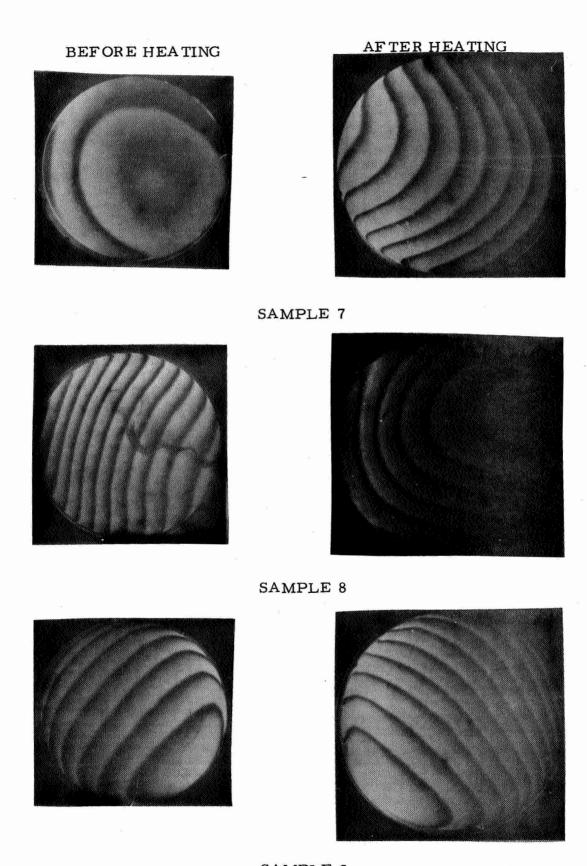
2.2.1.4 Hot Cycling Stability - Again all nine samples were cycled simultaneously in an oven. Each sample was protected with four layers of tissue and then all nine samples were wrapped with four (4) layers of tissue. The oven temperature was raised 50° per hour to 300° F and held four (4) hours. The oven temperature was then reduced at 50° per hour to 150° F and then turned off. The pieces were allowed to oven-cool overnight.

All three pieces as cycled above showed considerable change. Two pieces changed 1/2 λ concave while retaining their regularity; and the third changed 1/4 λ convex and went 1/2 λ astigmatic. Results are shown in Figure 6.

2.2.1.5 Stress Stability - The samples were subjected to a pressure differential of 14 psi supported on a soft rubber "O-ring", 4 inches in diameter, for two minutes. While subjected to the pressure differential, the samples were 7 or 8 λ concave. The stress cycling produced no change (less than $\lambda/8$) in figure in any of the three samples. Results are shown in Figure 7.

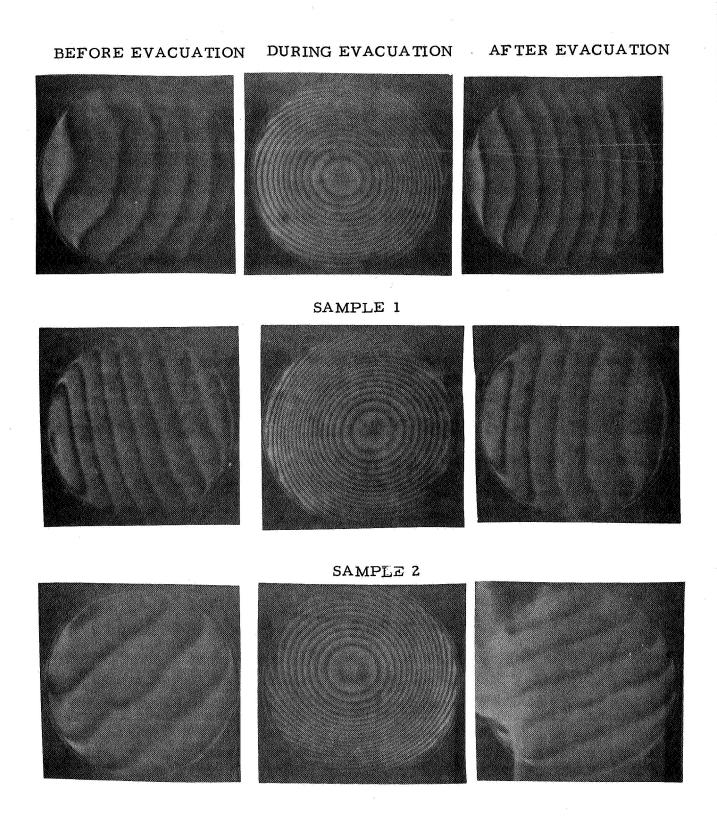
2.2.2 Beryllium

- 2.2.2.1 Reflectivity The reflectivity was measured in the same manner as the nickel and the results are shown in Table I and Figure 1. Scattering varied from 1/2 percent at 4000 Å to 1.8 percent at 6500 Å.
- 2.2.2 Time Stability One sample (#5) has remained unchanged for a year. Two samples (#4 and #6) were unchanged for three months, then changed from $\lambda/4$ cc flat and $\lambda/2$ regular to $\lambda/2$ cc flat and $\lambda/2$ regular between the third and seventh months, and then returned to their original figure between the seventh and twelfth months. Results are shown in Table II and Figures 8, 9 and 10.



SAMPLE 9

Figure 6. Hot Cycling Stability Testing Of Nickel Samples



SAMPLE 3

Figure 7. Stress Stability Testing of Nickel Samples.

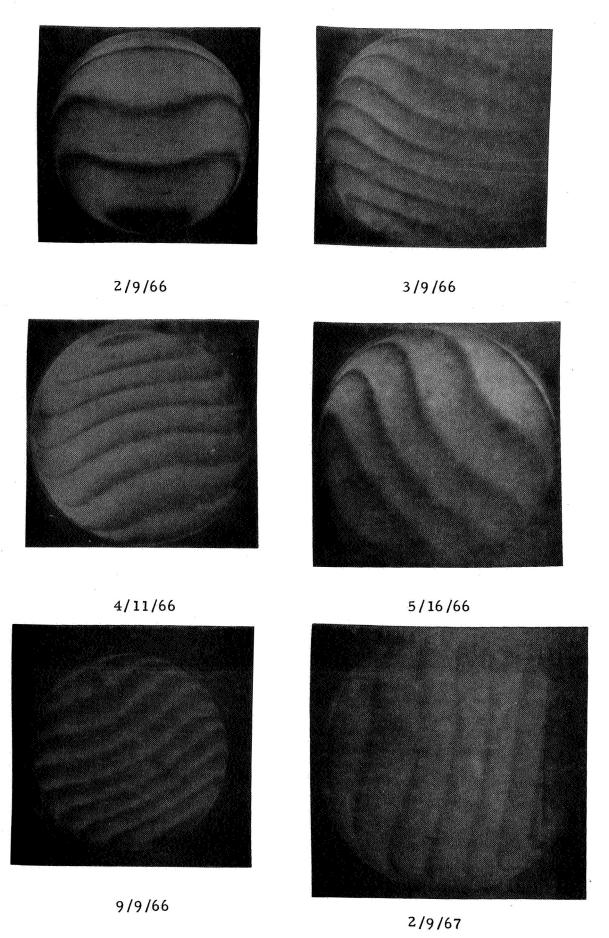
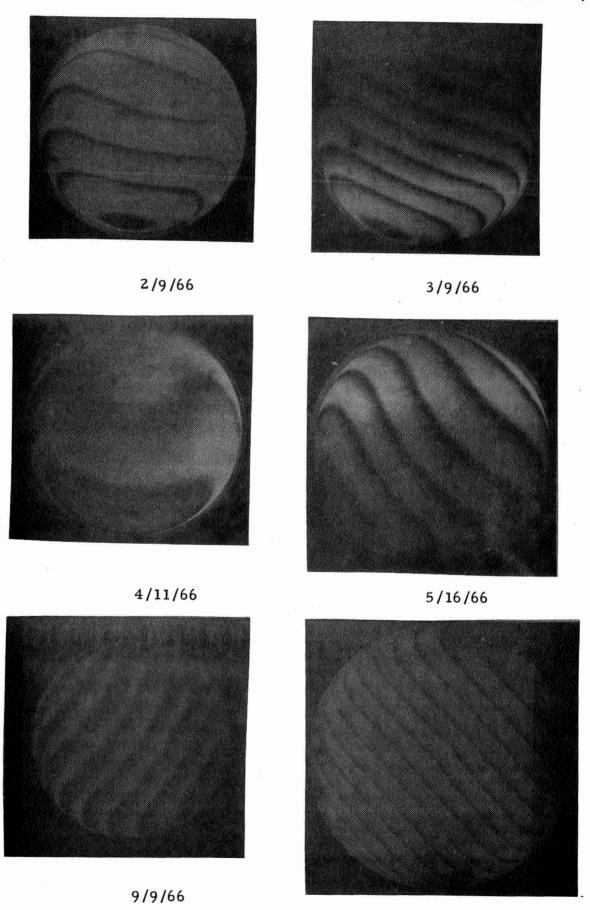


Figure 8. Time Stability Testing Of Beryllium (Sample No. 4)



2/9/67
Figure 9. Time Stability Testing of Beryllium (Sample No. 5)

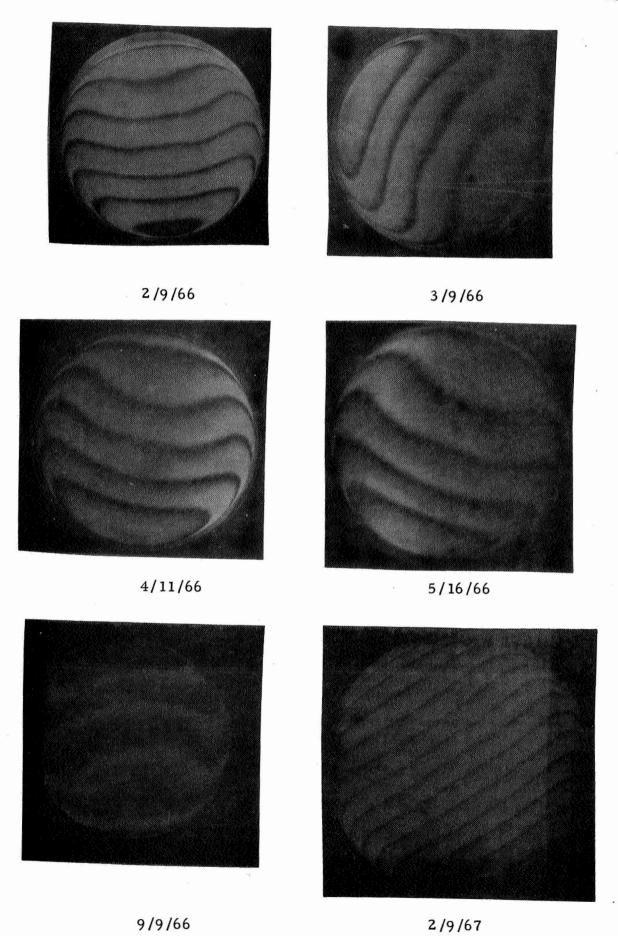
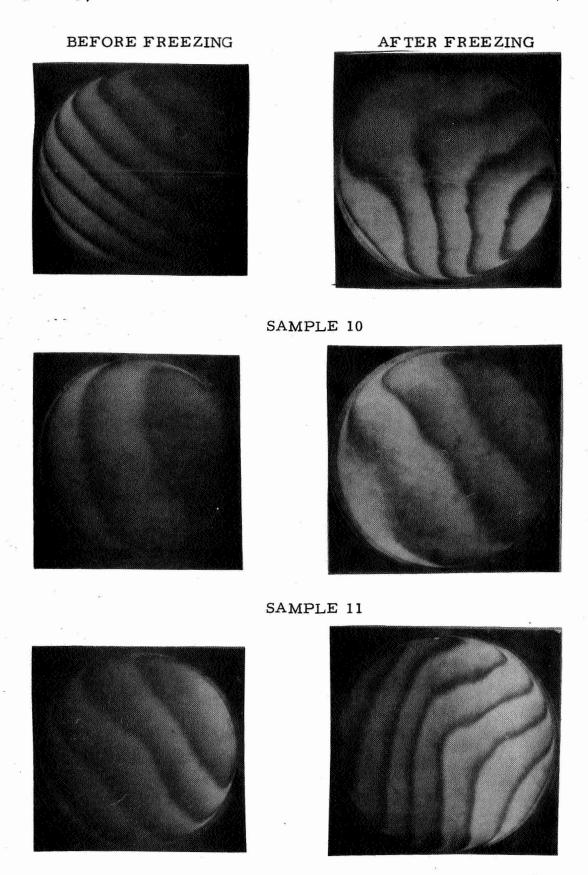


Figure 10. Time Stability Testing Of Beryllium (Sample No. 6)

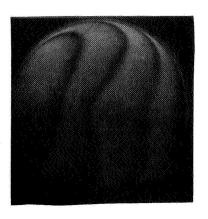
- 2.2.2.3 Cold Cycling Stability The beryllium samples were cycled as were the nickel. Two of the samples remained unchanged (less than λ/8); the third (Be-10) changed 1/4 λ concave and 1/4 λ regular to 1/2 λ concave, 1 λ regular and 1 λ astigmatic. Results are shown in Figure 11.
- 2.2.2.4 Hot Cycling Stability The beryllium samples were cycled as were the nickel. Two of the samples remained unchanged (less than λ /8); the third (Be-9) changed from 1/2 λ concave and 1/2 λ regular to 1 λ concave and 1/2 λ regular. Results are shown in Figure 12.
- 2.2.2.5 Stress Stability The beryllium samples were cycled as were the nickel. The stress cycling produced no change (less than $\chi/8$) in figure in any of the three samples. Results are shown in Figure 13.
- 2.2.3 Haynes 25
- 2.2.3.1 Reflectivity The reflectivity was measured in the same manner as the nickel and beryllium, and the results are shown in Table I and Figure 1. Scattering varied from 2.3 percent to 3.1 percent and probably averaged less than 2.7 percent.
- 2.2.3.2 Time Stability One sample (#6) has remained unchanged for one year. One sample (#5) changed from $\lambda/4$ cc flat and $\lambda/2$ regular to $\lambda/2$ cc flat and $\lambda/2$ regular during the first month and has remained unchanged since. The third sample (#4) remained unchanged during the first three months but changed from $\lambda/4$ cc flat and $\lambda/2$ regular to $\lambda/4$ cc flat and $\lambda/4$ regular between the third and seventh months, and then returned to its original figure between the seventh and twelfth months. The results are shown in Table II and Figures 14, 15 and 16.



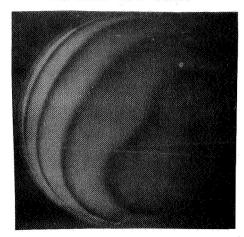
SAMPLE 12

Figure 11. Cold Cycling Stability Testing of Beryllium (Sample No. 12)

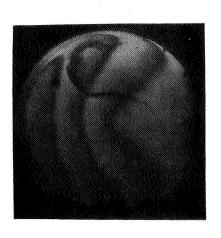
BEFORE HEATING

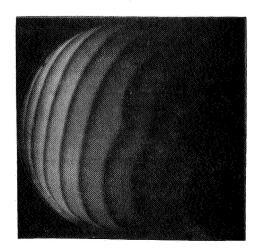


AFTER HEATING

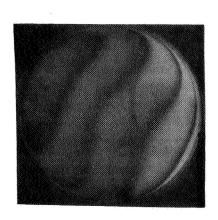


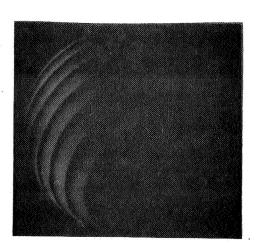
SAMPLE 7





SAMPLE 8





SAMPLE 9

Figure 12. Hot Cycling Stability Testing Of Beryllium Samples

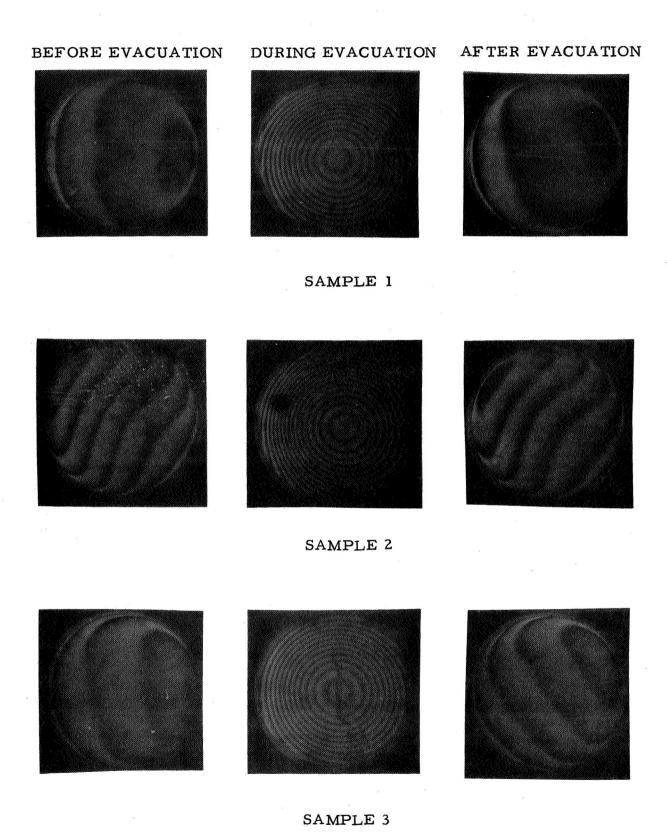


Figure 13. Stress Stability Testing Of Beryllium Samples

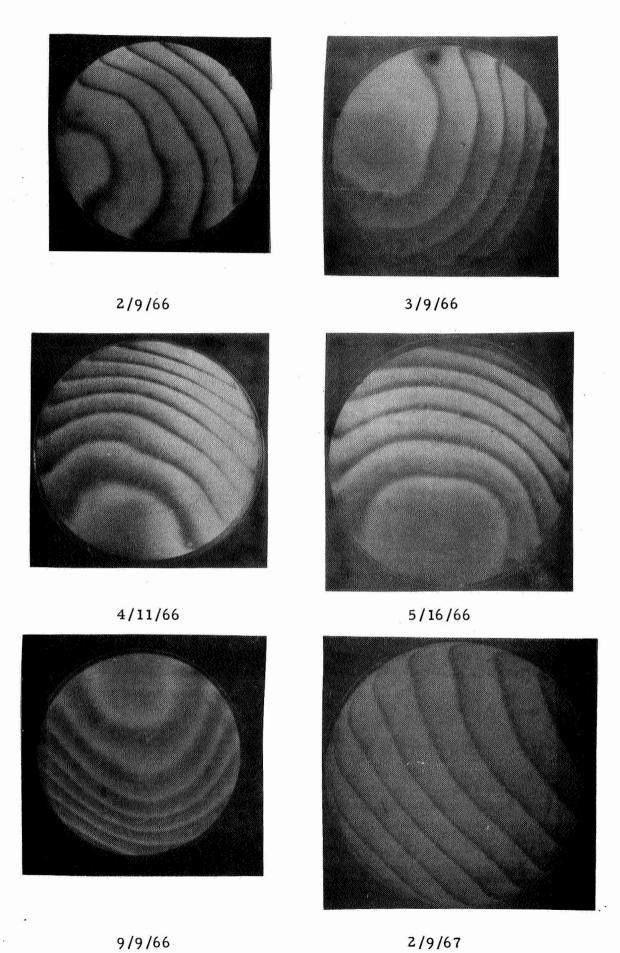


Figure 14. Time Stability Testing Of Haynes 25 (Sample No. 4)

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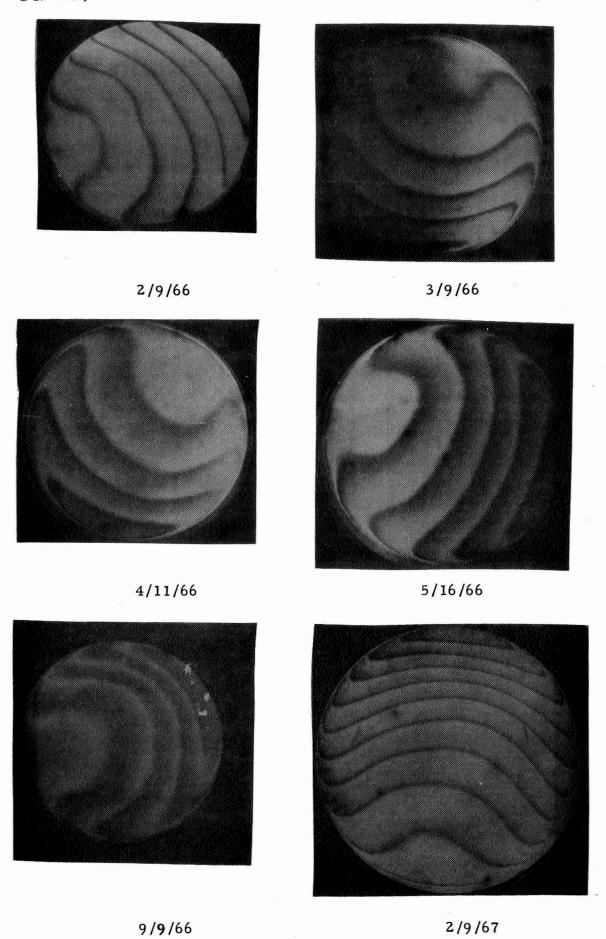


Figure 15. Time Stability Testing of Haynes 25 (Sample No. 5)

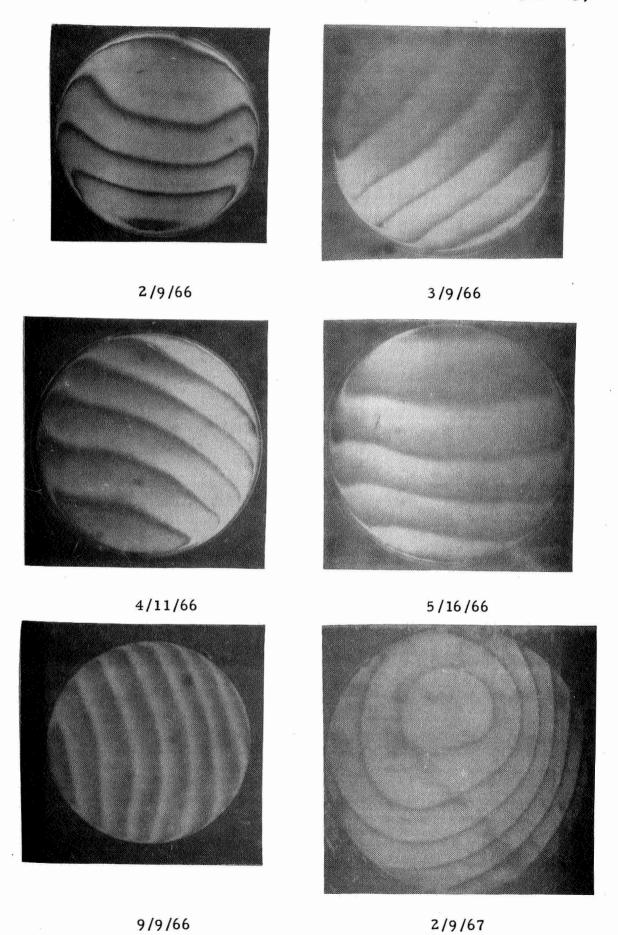


Figure 16. Time Stability Testing Of Haynes 25 (Sample No. 6)

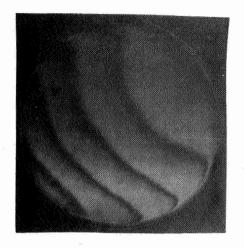
- 2.2.3.3 Cold Cycling Stability The Haynes 25 samples were cycled as were the nickel. Two of the samples remained unchanged (less than λ/8); the third sample remained unchanged in power and regularity but went λ/4 astigmatic. Results are shown in Figure 17.
- 2.2.3.4 Hot Cycling Stability The Haynes 25 samples were hot cycled as were the nickel. All three samples changed considerably. The first changed from $\lambda/2$ flat and regular to 2-1/2 λ concave, 1 λ regular and $\lambda/2$ astigmatic. The other two remained unchanged in power and regularity but went 1 λ and 1-1/2 λ astigmatic. Results are shown in Figure 18.
- 2.2.3.5 Stress Stability The Haynes 25 samples were cycled as were the nickel. All three samples remained unchanged (less than $\chi/8$). Results are shown in Figure 19.

3.0 CONCLUSIONS AND RECOMMENDATIONS

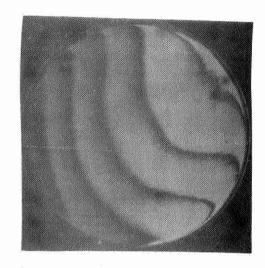
1. It has been concluded that the Lurium 5, the Aluminum 2024, the Aluminum 6061, and Lockalloy cannot, at this time, be optically polished and figured to better than $\lambda/2$. Techniques which produce either a clean surface or a good figure have been found, but no technique which produces both has been found.

It can be concluded from the difficulty in achieving a clean surface with the blocks of beryllium samples that the use of this material might be restricted to flats and spheres. If an asphere were to be figured, "contact" necessarily would be poor and a poor surface might result as with block.

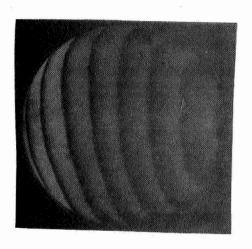
BEFORE FREEZING

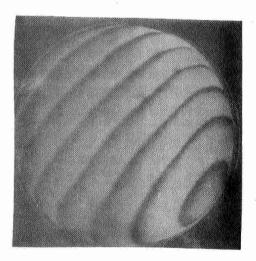


AFTER FREEZING

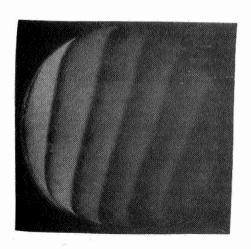


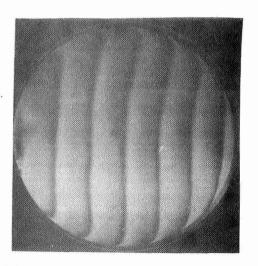
SAMPLE 11





SAMPLE 12



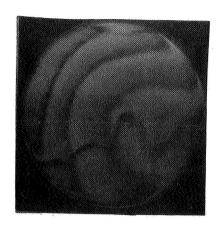


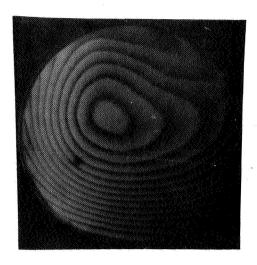
SAMPLE 13

Figure 17. Cold Cycling Stability Testing Of Haynes 25 Samples.

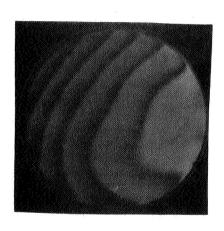
BEFORE HEATING

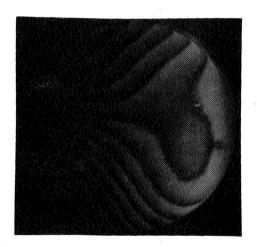
AFTER HEATING



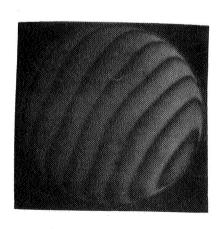


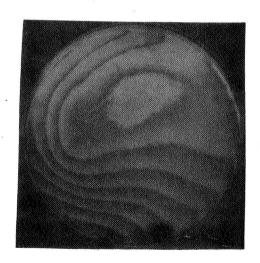
SAMPLE 7





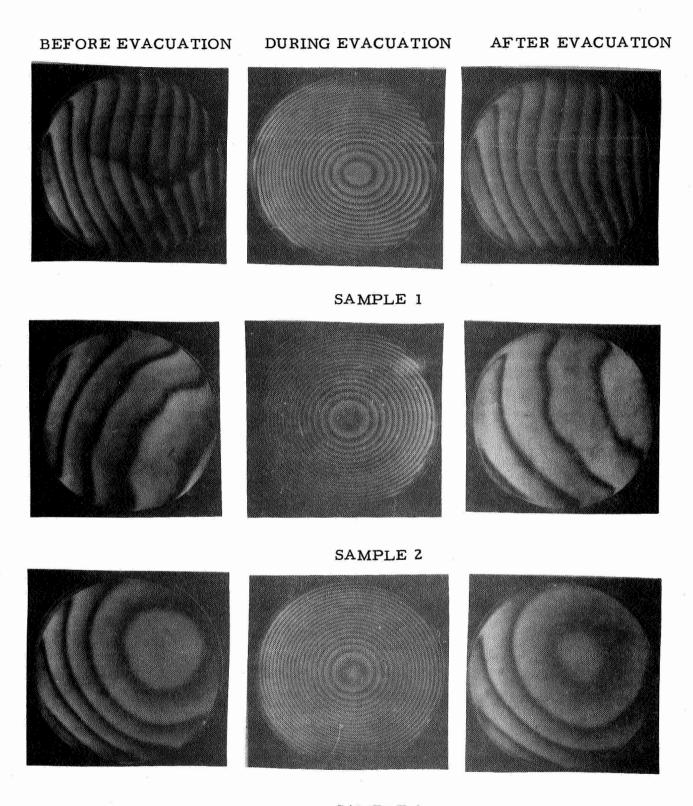
SAMPLE 8





SAMPLE 9

Figure 18. Hot Cycling Stability Testing of Haynes 25 Samples



SAMPLE 3

Figure 19. Stress Stability Testing Of Haynes 25 Samples

As can be seen from Table I and Figure 1, all three metals are inferior in reflectivity to evaporated aluminum on an optically polished glass substrate. This deficiency can be corrected by aluminizing the metals. However, the scattering (total reflectivity minus specular reflectivity) is much higher than for optically polished glass substrates. These comparative figures are:

Glass - < .5%

Beryllium - .5 to 1.8%

Haynes 25 - 2.3 to 3.1%

Nickel - 2.5%

Such scattering would cause contrast reduction and might limit the uses of these metals to less acute systems.

- 3. All three materials have shown sufficient change with time to make their use as precision optical surfaces questionable Beryllium appears to be the best of the three for time stability.
- 4. One sample of each of the three materials changed in figure during the cold cycling. There is little difference between the three in this respect. The suitability of any of these materials would depend upon the precision required after cycling.
- 5. The beryllium is judged to be the most suitable material during hot cycling. Only one of the samples changed figure and then only by $\lambda/2$. All of the nickel and the Haynes 25 samples showed more change than the one beryllium sample which changed. The nickel showed less than 1/2 the change of the Haynes 25. Again,

the suitability of any of these materials would depend upon the precision required after cycling.

6. All of the materials are satisfactory with respect to stress stability.

4.0 NEW TECHNOLOGY

No new technology has been reduced to practice during this contract.